

WORLD INTELLECTUAL PROPERTY ORGANIZATION ORGANISATION MONDIALE DE LA PROPRIÉTÉ INTELLECTUELLE

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PATENT COOPERATION TREATY (PCT) TRAITÉ DE COOPÉRATION EN MATIÈRE DE BREVETS (PCT)

CERTIFIED COPY OF THE INTERNATIONAL APPLICATION AS FILED AND OF ANY CORRECTIONS THERETO

COPIE CERTIFIÉE CONFORME DE LA DEMANDE INTERNATIONALE, TELLE QU'ELLE A ÉTÉ DÉPOSÉE, AINSI QUE DE TOUTES CORRECTIONS Y RELATIVES

International Application No. Demande internationale n° PCT/IB01/01018

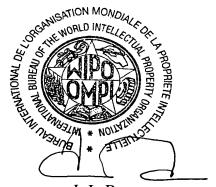
International Filing Date
Date du dépôt international

11 June 2001
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Geneva/Genève.

14 November 2003 (14.11.03) International Bureau of the World Intellectual Property Organization (WIPO)

Bureau International de l'Organisation Mondiale de la Propriété Intellectuelle (OMPI)



J.-L. Baron

Head, PCT Receiving Office Section Chef de la section "office récepteur du PCT"

PCT REQUEST

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| 0 | For receiving Office use only | |
|---------|---|--|
| 0-1 | International Application No. | PCT / IB 0 1 / 0 1 0 1 8 |
| 0-2 | International Filing Date | (1 1. 05. 01) |
| 0-3 | Name of receiving Office and "PCT International Application" | INTERNATIONAL BUREAU OF WIPO PCT International Application |
| 0-4 | Form - PCT/RO/101 PCT Request | |
| 0-4-1 | Prepared using | PCT-EASY Version 2.91 (updated 01.01.2001) |
| 0-5 | Petition | |
| | The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty | |
| 0-6 | Receiving Office (specified by the applicant) | International Bureau of the World Intellectual Property Organization (RO/IB) |
| 0-7 | Applicant's or agent's file reference | 5520-PCT |
| 1 | Title of invention | STABLE TRANSPARENT PERFUMING EMULSION |
| 11 | Applicant | |
| II-1 | This person is: | applicant only |
| II-2 | Applicant for | all designated States except US |
| 11-4 | Name | FIRMENICH SA |
| II-5 | Address: | 1, route des Jeunes P.O. Box 239 CH-1211 GENEVA 8 Switzerland |
| 11-6 | State of nationality | СН |
| II-7 | State of residence | СН |
| II-8 | Telephone No. | +41 22 780 22 11 |
| 11-9 | Facsimile No. | +41 22 780 33 38 |
| II-10 | e-mail | ANNICK.BOCCARD@FIRMENICH.COM |
| III-1 | Applicant and/or inventor | |
| 111-1-1 | This person is: | applicant and inventor |
| III-1-2 | Applicant for | US only |
| III-1-4 | Name (LAST, First) | STORA, Thierry |
| III-1-5 | Address: | 77, place de la Mairie F-01630 SERGY |
| | | France |
| III-1-6 | State of nationality | FR |
| 111-1-7 | State of residence | FR |

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| Interpretation between population and the interpretation of the competent international Authorities as: IV-1-1 IV-1-2 Address: IV-1-1 IV-1-2 Address: IV-1-3 Telephone No. IV-1-4 Telephone No. IV-1-4 Telephone No. IV-1-4 Designation of States IV-1 Obegination of States IV-1 Obe | IV-1 | Ag nt or c mm n representative; or address for c rrespondence | | | |
|--|--------|---|--|-----------------------------|--|
| Number N | | behalf of the applicant(s) before the | agent | agent | |
| IV-1-2 Address: FIRMENICH SA P.O. Box 239 CH-1211 GENEVA 8 Switzerland H41 22 780 22 11 H41 22 780 33 38 IV-1-4 | IV-1-1 | 1 ' | SALVATERRA-GARCIA. Maria de Lurdes | | |
| P.O. Box 239 CH-1211 GENEVA 8 Switzerland H41 22 780 22 11 H41 22 780 33 38 | IV-1-2 | Address: | 1 | | |
| IV-1-3 Telephone No. | | | | | |
| IV-1-3 Telephone No. +41 22 780 22 11 +41 22 780 33 38 V | | | CH-1211 GENEVA 8 | | |
| V-1-4 Facsimile No. | | | Switzerland | | |
| V-1 Profit Sates V-1 Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) V-2 National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) V-2 National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) V-5 Precautionary Designation Statement in addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designations are subject to confirmation and that any designation of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limits is to be regarded as withdrawn by the applicant at the expiration of that time limits. V-6 Exclusion(s) from precautionary designations VI Priority claim NONE VIII Check list number of sheets electronic file(s) attached VIII Check list number of sheets electronic file(s) attached VIII-2 Description 14 VIII-3 Claims 2 VIII-4 Abstract 1 EZABST00.TXT | IV-1-3 | Telephone No. | +41 22 780 22 11 | | |
| Regional Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) The patenth of the European Patent Convention and the European Patent Office (EPO) (ISA/EP) Viii Check list None European Patent Office (EPO) (ISA/EP) Viii Check list number of sheets electronic file(s) attached VIII-1 Request 3 - VIII-2 Description 14 - VIII-3 Claims 2 - VIII-4 Abstract 1 EZABSTOO.TXT VIII-5 Drawings 0 - | IV-1-4 | Facsimile No. | +41 22 780 33 38 | | |
| Cother kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) State which is a Contracting State of the European Patent Convention and of the PCT | V | Designation of States | | | |
| if any, are specified between parentheses after the designation(s) concerned) V-2 National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) V-5 National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) V-6 Precautionary Designation Statement In addition to the designation smade under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation of of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. V-6 Exclusion(s) from precautionary designations VI Priority claim NONE VII-1 International Searching Authority Chosen VIII-1 Check list number of sheets electronic file(s) attached VIII-1 Request 3 VIII-2 Description 14 VIII-3 Claims 2 VIII-4 Abstract 1 EIT IN MC NL PT SE TR and any other state of the European Patent Convention and of the European Patent Convention and of the European Patent Office (EPO) (ISA/EP) Chosen VIII-1 Priority claim NONE State which is a Contracting State of the European Patent Office (EPO) (ISA/EP) Chosen VIII-1 Check list number of sheets electronic file(s) attached VIII-1 Provings 0 | V-1 | | | | |
| concerned) the European Patent Convention and of the PCT National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) V-5 Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. V-6 Exclusion(s) from precautionary designations VI Priority claim NONE VII-1 International Searching Authority Chosen NONE Priority claim International Searching Authority Chosen NONE Description 14 Pescription 14 Pescription 14 Pescription 15 Patent Convention and of the VII-1 Proceedings of the State(s) in the priority claim of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. Priority claim NONE VII-1 International Searching Authority Chosen NONE Priority claim NONE Proceedings of the State(s) and the proceedings of the State(s) attached VIII-1 Request 3 VIII-1 Pescription 14 Pescription 14 Pescription 1- Pescription 1- Pescription 1- Provings Patent Convention and of the transport of the transport of the proceedings of the | | | | _ | |
| V-2 National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) V-5 Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation with those additional declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. V-6 Exclusion(s) from precautionary designations VI Priority claim NONE VIII-1 International Searching Authority Chosen number of sheets electronic file(s) attached VIII-1 Check list number of sheets electronic file(s) attached VIII-2 Description 14 | | 1. | 1 | - | |
| V-2 National Patent (other kinds of protection or treatment, if any, are specified between parentheses after the designation(s) concerned) V-5 Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation which would be permitted under the PCT except any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. V-6 Exclusion(s) from precautionary designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. V-7 V-8 V-9 V- | | Concerned) | - | Convention and of | |
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| V-5 Precautionary Designation Statement In addition to the designations made under items V-1, V-2 and V-3, the applicant also makes under Rule 4.9(b) all designations which would be permitted under the PCT except any designation(s) of the State(s) indicated under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. V-6 | | | | | |
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| under item V-6 below. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. V-6 | | permitted under the PCT except any | | | |
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| VI Priority claim NONE VII-1 International Searching Authority Chosen European Patent Office (EPO) (ISA/EP) VIII Check list number of sheets electronic file(s) attached VIII-1 Request 3 - VIII-2 Description 14 - VIII-3 Claims 2 - VIII-4 Abstract 1 EZABST00.TXT VIII-5 Drawings 0 - | V-6 | Exclusion(s) from precautionary | NONE | | |
| VIII Chosen Image: Chosen of the choice of | VI | | NONE | | |
| VIII-1 Request 3 - VIII-2 Description 14 - VIII-3 Claims 2 - VIII-4 Abstract 1 EZABST00.TXT VIII-5 Drawings 0 - | VII-1 | | European Patent Offi | ce (EPO) (ISA/EP) | |
| VIII-2 Description 14 — VIII-3 Claims 2 — VIII-4 Abstract 1 EZABST00.TXT VIII-5 Drawings 0 — | | Check list | The state of the s | electronic file(s) attached | |
| VIII-3 Claims 2 - VIII-4 Abstract 1 EZABST00.TXT VIII-5 Drawings 0 - | | · | | | |
| VIII-4 Abstract 1 EZABST00.TXT VIII-5 Drawings 0 - | | Description | 14 | _ | |
| VIII-5 Drawings 0 - | | Claims | 2 | | |
| | | Abstract | 1 | EZABST00.TXT | |
| VIII-7 TOTAL 20 | | | | <u> </u> | |
| | VIII-7 | TOTAL | 20 | | |

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| | Accompanying items | paper document(s) attached | electronic file(s) attached |
|--|--|----------------------------|-----------------------------|
| VIII-8 | Fee calculation sheet | ✓ | 1- |
| VIII-9 | Separate signed power of attorney | ¥ | 1- |
| VIII-16 | PCT-EASY diskette | 1- | diskette |
| VIII-18 | Figure of the drawings which should accompany the abstract | | |
| VIII-19 | Language of filing of the international application | English | <i>y</i> |
| IX-1 | Signature of applicant or agent | | has |
| IX-1-1 | Name (LAST, First) | SALVATERRA-GARCIA, N | Maria de Lurdes |
| | 5 4 5 4 5 | / | |
| 10-1 | Date of actual receipt of the | 1 | (4) 4 62 44 |
| | purported international application | 11 JUNE 2001 | 1 1, 06, 01 |
| 10-2 | purported international application Drawings: | 11 JUNE 2001 | 1 1, 05, 01 |
| 10-2 | purported international application | 11 JUNE 2001 | 1 1. 05. 01 |
| 10-2 10-2-1 | purported international application Drawings: | 11 JUNE 2001 | 1 1, 06, 01 |
| 10-1 10-2 10-2-1 10-2-2 10-3 | purported international application Drawings: Received | 11 JUNE 2001 | 1 1, 06, 01 |
| 10-2 10-2-1 10-2-2 10-3 | purported international application Drawings: Received Not received Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application Date of timely receipt of the required corrections under PCT Article 11(2) | 11 JUNE 2001 | 1 1, 05, 01 |
| 10-2 10-2-1 10-2-2 10-3 | purported international application Drawings: Received Not received Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application Date of timely receipt of the required | 11 JUNE 2001 ISA/EP | 1 1, 05, 01 |

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| 11-1 | Date of receipt of the record copy by | |
|------|---------------------------------------|--|
| | the International Bureau | |

PCT (ANNEX - FEE CALCULATION SHEET)
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(This sheet is not part of and does not count as a sheet of the international application)

| 0 | For receiving Office use only | | | |
|-------------|--|-----------------------|---------------------------------------|--------------|
| 0-1 | International Application No. | 207112 | | |
| | | PUI/IB | 01/01018 | |
| 0-2 | Date stamp of the receiving Office | 1 1 | JUNE 2001 | |
| | | <u> </u> | | |
| 0-4 | Form - PCT/RO/101 (Annex) | | | |
| 0-4-1 | PCT Fee Calculation Sheet | DOD | ÷ 0 01 | |
| 0-4-1 | Prepared using | PCT-EASY Vers | | |
| 0.0 | Applicant's or agent's file reference | (updated 01.0 | 1.2001) | |
| 0-9 | | 5520-PCT | · · · · · · · · · · · · · · · · · · · | |
| 2 | Applicant | FIRMENICH SA, | | |
| 12 12-1 | Calculation of prescribed fees Transmittal fee T | fee amount/multiplier | total amounts (CHF) | |
| | Search fee S | | | |
| 12-2 | | ₽ | 1'460 | |
| 12-3 | International fee | | | |
| | Basic fee | | | |
| | (first 30 sheets) b1 | 650 | | |
| 12-4 | Remaining sheets | 0 | | |
| 12-5 | Additional amount (X) | 15 | | |
| 12-6 | Total additional amount b2 | 0 | | |
| 12-7 | b1 + b2 = B | 650 | | |
| 12-8 | Designation fees | | | |
| | Number of designations contained in international application | 8 | | |
| 12-9 | Number of designation fees payable (maximum 6) | 6 | | |
| 12-10 | Amount of designation fee (X) | 140 | | |
| 12-11 | Total designation fees D | 0 | | |
| 12-12 | PCT-EASY fee reduction R | -200 | | |
| 12-13 | Total International fee (B+D-R) | ₽ | 450 | |
| 12-17 | TOTAL FEES PAYABLE (T+S+I+P) | ₽ | 2'010 | |
| 12-18 | Designation fees are not paid at this time | √ | | |
| 12-19 | Mode of payment | authorization | to charge dep | osit account |
| 12-20 | Deposit account instructions | | | |
| | The receiving Office: | International | Bureau of the | World |
| | | Intellectual 1 | Property Organ | ization |
| | | (RO/IB) | | |
| 12-20- 1 | is hereby authorized to charge the total fees indicated above to my deposit | ✓ | | |
| 12-20- | account is hereby authorized to charge any | √ | | |
| 2 | deficiency or credit any over-payment in the total fees indicated above to my deposit account | • | | |
| 12-20- 3 | is hereby authorized to charge the fee for preparation and transmittal of the priority document to the International Bureau of WIPO to my deposit account | √ | | |

PCT (ANNEX - FEE CALCULATION SHEET)
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5520-PCT

| 12-21 | Deposit account No. | 11557 |
|-------|---------------------|------------------------------------|
| 12-22 | Date | 11 June 2001/(1/1.06.2001) |
| 12-23 | Name and signature | SALVATERRA-GARCIA, Maria de Lurdes |

VALIDATION LOG AND REMARKS

| 13-2-2 | Validation messages States | Green? More designations could be made. The | |
|--------|-------------------------------|---|--|
| | | following States have not been | |
| | | designated: AP: (GH, GM, KE, LS, MW, MZ, | |
| | | SD, SL, SZ, TZ, UG, ZW); EA: (AM, AZ, | |
| | | BY, KG, KZ, MD, RU, TJ, TM); OA: (BF, | |
| | | BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, | |
| | | | |
| | | NE, SN, TD, TG); AE, AG, AL, AM, AT, AU, | |
| | | AZ, BA, BB, BG, BY, BZ, CH, LI, CR, CU, | |
| | İ | CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, | |
| | | GE, GH, GM, HR, HU, ID, IL, IS, KE, KG, | |
| | | KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, | |
| | | MA, MD, MG, MK, MN, MW, MZ, NO, NZ, PL, | |
| | | PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, | |
| | | TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, | |
| | | ZW. Please verify. | |
| 13-2-3 | Validation messages Names | Green? | |
| | Names | Agent 1.: Where several first/given | |
| | | names are indicated, they should | |
| | | preferably be separated by a comma. | |
| | | Please verify. | |
| 13-2-4 | Validation messages Priority | Green? | |
| | | No priority of an earlier application | |
| | | has been claimed. Please verify | |
| 13-2-6 | Validation messages | Green? | |
| | Contents | The international application contains | |
| | | no drawings. Please verify. | |
| 13-2-7 | Validation messages | Green? | |
| | Fees | Designation Fees not paid: see Help for | |
| | | payment time limits. | |
| 13-2-8 | Validation messages | Green? | |
| | Payment | Please ensure that you have a valid | |
| | | deposit account with the receiving | |
| | | Office selected. | |
| | <u> </u> | | |

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Stable transparent perfuming emulsion

Technical Field

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The present invention relates to the field of perfumery. It concerns more particularly an alcohol-free perfuming composition in the form of a fluid, transparent water-in-oil or oil-in-water emulsion. The emulsion of the invention is characterised by an outstanding stability, the densities of the respective oily and aqueous phases being particularly close to one another.

Background of the Invention

Several criteria must be met in a well-formulated emulsion. Two of the most readily apparent requirements are that the emulsion possesses adequate physical and chemical stability.

First of all, chemical stability of the various components of the emulsion should receive a particular attention in perfumery, since the main materials used in perfuming compositions, namely perfuming ingredients, are well-known to be particularly difficult to stabilise in formulations such as emulsions (Cosmetics and Toileteries[®] Magazine, Vol. 109, pages 71-75, 1994). The stabilisation of formulations comprising high fragrance levels such as those concerned by the present invention constitutes therefore a specific and challenging problem, notably different from that of the stabilisation of a cosmetic emulsion, which comprises other kinds of components, the nature of which is generally dependent on the type of the final cosmetic product (typical active ingredients include natural or synthetic oils, hydrocarbons, halogenated carbons, esters of mineral acids, silicones). Furthermore, such cosmetic compositions contain very low amounts of perfuming ingredients.

On the other hand, an essential requirement sought in the formulation of an emulsion of any kind is the physical stability, without which the emulsion will soon revert back to separate bulk phases. One typical phenomenon associated with physical instability is constituted by the upward and downward movement of dispersed droplets relative to the continuous phase, termed creaming or sedimentation respectively. The factors that

influence the rate of sedimentation or creaming are the diameter of the suspended droplets, the viscosity of the suspending medium and the difference in densities between the dispersed phase and the dispersion medium.

The prior art discloses a first means to avoid this destabilisation phenomenon, consisting in reducing the particle size of the dispersed system, which contributes greatly toward overcoming or minimising creaming.

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Therefore, perfuming compositions in the form of microemulsions are known from the prior art. Microemulsions are liquid dispersions of water and oil that are made homogeneous, transparent and stable by the addition of relatively large amounts of a surfactant and a co-surfactant. While emulsions are unstable, microemulsions are stable and, therefore, they are formed spontaneously when oil, water, surfactants and co-surfactants are mixed together. Conversely, emulsions require input of considerable mechanical energy for their preparation. Furthermore, microemulsion droplets have a narrow droplet size distribution with a mean diameter range of approximately 5-50 nm. Consequently this type of formulation scatters little light and is therefore transparent or at least translucent, while emulsions have very broad droplet size distributions and, as the emulsion droplets are comparable in size or larger than the wavelength of visible light, they scatter it more or less strongly depending on the difference in refractive index between oil and water. Thus most emulsions are opaque. It appears therefore that emulsions and microemulsions constitute two distinct dispersed systems, of different nature for the reasons here-above mentioned.

Now, for the purpose of the present invention, it is not desirable to incorporate an important amount of surfactant relative to the amount of perfuming ingredients, as this limits considerably the amount of fragrance that can be incorporated in the mixture. Therefore, a microemulsion does not constitute a suitable dispersed system for the present invention, which has to be in the form of an emulsion.

Another known transparent dispersed system of this type is the nanoemulsion characterised by an average size of the oily phase droplets below ca. 30-75 nm. The droplets are small enough to make the emulsion translucent or partially transparent. Although these nanoemulsions present the advantage of needing lesser amount of surfactants than the microemulsions, they present nevertheless the disadvantage that their process of preparation is often difficult and delicate, and thus costly.

The other manner disclosed in the prior art to stabilise a conventional emulsion is the addition of a polymer to the dispersed phase. However, this solution presents the

drawback of thickening said phase, thus increasing the viscosity of the emulsion such that it is no longer easily sprayable.

WO 00/33804, the contents of which is hereby included by reference, which describes a perfuming composition in the form of a transparent emulsion, already matches most of the criteria required for such an emulsion (transparency, fluidity). However, the emulsion there-described has now been improved in terms of physical stability, thanks to a novel solution to the physical stability problem here-above described.

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In fact, the invention now provides a transparent, fluid, water-in-oil or oil-in-water emulsion characterised by the fact that the densities of the respective continuous and dispersed phases have almost matching values. This solution to the well-known problem of physical instability, and more particularly of creaming and sedimentation, specifically adapted to a dispersed system of the emulsion type, has been achieved thanks to the presence of a specific additive in the oily phase of the emulsion, namely a volatile fluorinated oil having a density higher than 1.

This class of compounds has already been used in some kinds of emulsions. In particular, EP 1064929 discloses the use of such a volatile fluorinated oil in a cosmetic composition for attenuating skin deficiencies and for giving a matt skin appearance. The cosmetic composition there-disclosed is not only totally distinct, from a formulation point of view, from a perfuming composition, but also the fluorinated oil is used in this prior art for a technical effect consisting in modifying the skin appearance, thus an aim distinct from that achieved by the present invention, i.e. the stabilisation of an emulsion.

EP 1064989 also discloses a cosmetic emulsion comprising a volatile fluorinated oil. Once again, the latter is described as being useful for its topical properties in such cosmetic applications, providing compositions of easy application, as well as fresh and soft products. Moreover, this document specifies that the fluorinated oil is very difficult to stabilise and may provoke an instability phenomenon such as foaming, demixing and changes in viscosity, and that it has thus to be used in combination with a specific surfactant, able to stabilise the emulsion. According to the teaching of this document, a volatile fluorinated oil is thus very difficult to stabilise.

It was therefore all the more surprising that we have now discovered, in a totally unexpected manner considering the teaching of the prior art, that a perfuming composition, in the form of an emulsion, could be advantageously stabilised by minimising the difference existing between the densities of the respective continuous and dispersed phases and that such an effect could be achieved by adding to the oily phase of the

emulsion a volatile fluorinated oil having a density higher than one, without any need of combining said ingredient with a specific surfactant such as disclosed in EP 1064989.

Together with the transparency, fluidity and stability requirements, the composition of the invention is also alcohol-free, which is an advantage henceforth sought in such formulations.

Description of the Invention

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It is thus an object of the present invention to provide an alcohol free perfuming composition in the form of a transparent water-in-oil or oil-in-water emulsion with an outstanding stability, characterised by the fact that the difference between the density of the oily phase and that of the aqueous phase in less than or equal to 0.005, preferably less than or equal to 0.003.

The emulsion of the invention is advantageously particularly resistant to phenomena such as creaming and sedimentation and constitutes a novel, effective solution to the instability problems known in the prior art compositions.

Therefore, according to the invention, it has been established that a substance can be added to a perfuming composition which is an emulsion and provide the technical effect of bringing closer the densities of the respective phases of this emulsion. A volatile fluorinated oil having a density higher than 1 constitutes such a substance. Another object of the invention is thus provided by a perfuming composition in the form of an emulsion, wherein the oily phase comprises a certain amount of a volatile fluorinated oil having a density higher than 1. And furthermore, the invention also concerns the use of said ingredient as a stabilisation agent in an emulsion.

The perfuming composition of the invention is in the form of a transparent water-in-oil or oil-in-water emulsion susceptible of being sprayed, comprising an aqueous phase and an oily phase containing at least 3% by weight of perfuming ingredients relative to the total weight of the composition, wherein the difference between the density of the oily phase and that of the aqueous phase is less than or equal to 0.005, preferably less than or equal to 0.003. This technical effect may be achieved when the oily phase contains a volatile fluorinated oil having a density higher than 1.

What is intended here by perfuming composition is a perfume vehicle, the main function of which is to impart, enhance or modify the odoriferous properties of a product.

In the case of an oil-in-water emulsion, the continuous phase is formed by water (aqueous phase) and the dispersed phase is formed by the oil (oily phase) which is essentially formed of perfuming ingredients. Conversely, the emulsion is a water-in-oil or inverse emulsion when the continuous phase is formed by the oil wherein the water is dispersed. The latter type of emulsion is preferred according to one embodiment of the invention.

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As mentioned above, the emulsion according to the present invention meets a certain number of criteria required in the field of perfumery, for applications such as perfumes, eaux de toilette, or Colognes.

First of all, the composition of the invention is alcohol free. There is at present a demand for such types of compositions, particularly from consumers with sensitive skins or whose skins are often exposed to the sun.

Moreover, the composition of the invention is fluid, or in other words susceptible of being sprayed. In particular, its viscosity is lower than 10 Pa.s, independently of the nature of the emulsion.

Furthermore, the emulsion is transparent, a desirable quality in the field of perfumery. The transparency is achieved thanks to the addition to the perfuming compositions of ingredients capable of modifying the refractive indices of the two phases, so as to form a transparent emulsion. The particular solid or liquid ingredients used to act either on the oily or on the aqueous phase in order to achieve transparency are described in WO 00/33804, the contents of which are hereby included by reference, namely as regards the citation of such ingredients.

The refractive index of a finished perfuming composition according to the invention has a value between ca. 1.40 and 1.44.

Now, besides all the mentioned criteria already met, the present invention brings an advantageous improvement to the known transparent emulsions. In fact, it has been discovered that the addition of a certain ingredient to the oily phase of the emulsion could significantly improve the physical stability of the emulsion, in particular with regard to the creaming and sedimentation problems. In particular, the addition of such an ingredient to the oily phase of the emulsion makes it possible to narrow the difference

between the respective densities of the two phases, so as to bring it within the limit defined above, namely lower or equal to 0.005, or even 0.003 in preferred embodiments.

The invention brings thus a novel solution to the known problem of physical stability, by providing means for acting on the relative densities of both phases.

We have observed that in the context of this invention it is possible to use a certain number of substances or agents having the capacity of acting on the oily phase as desired and the choice of which depends on individual criteria that can be easily established by the skilled person for each composition. The above-mentioned agent which will be used to act on the oily phase must be soluble in the latter.

Preferably the agent is chosen with a density higher than 1.

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It has been discovered that the desired here-above described technical effect could be advantageously achieved in particular by adding to the oily phase a volatile fluorinated oil having a density higher than 1. The technical effect obtained with this compound is particularly surprising considering that, amongst the prior art, EP 1064989 teaches the need to combine said ingredient with a particular surfactant in order to stabilise it in an emulsion.

Preferably, the fluorinated oil used according to the present invention is selected from the group consisting of hydrofluoroethers such as methoxynonafluorobutane or ethoxynonafluorobutane. In a particular embodiment the ingredient added to the oily phase of the emulsion is methoxynonafluorobutane. The volatile fluorinated oil may also be used in mixture with a less volatile fluorinated oil such as a perfluoroalcane. Perfluorodecaline is an example of a suitable perfluoroalcane for this purpose.

The amount of fluorinated oil added to the oily phase is comprised between 5 and 50% by weight relative to the oily phase.

The emulsions of the invention can contain from 5 to 50% by weight, preferably from 10 to 35% by weight, of dispersed phase and from 50 to 95% by weight, preferably 65 to 90% by weight, of continuous phase. These values are relative to the total weight of the emulsion and are independent of the fact that the compositions of the invention are in the form of a water-in-oil or an oil-in-water emulsion.

The oily phase is essentially formed of perfuming ingredients. Typically it comprises from 15 to 60%, preferably from 20 to 50% by weight of perfuming ingredients.

The aqueous phase comprises from 20 to 65% of water.

Optionally, the composition of the invention may further contain at least one surfactant. This surfactant will be used in a proportion of 0 to 8%, preferably 0.1 to 5% by

weight, relative to the total weight of the emulsion. The best results were obtained using 2 to 5% by weight of surfactant.

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Different types of surfactants can be used in the context of the invention. One can mention non-ionic, cationic, anionic, the amphoteric surfactants and the phospholipids, which may all be used in the present invention. Preferably, there is used a non-ionic surfactant or a mixture of two non-ionic surfactants. As non restrictive examples, the ethoxylated nonylphenol comprising 9 to 10 units of ethyleneglycol (on sale under the name of Triton® N-101; origin: Fluka, Switzerland) or the ethoxylated undecanol comprising 8 units of ethyleneglycol (on sale under the name of Imbentin® 0800; origin: Kolb AG, Switzerland) can be cited. Other examples include the surfactants known under the tradename Tween® (origin: ICI, England), such as Tween® 20 [polyoxyethylene (20) sorbitan monolaurate], Tween® 40 [polyoxyethylene (20) sorbitan monopalmitate], Tween® 80 and sorbitan monostearate] Tween[®] 60 (20)[polyoxyethylene [polyoxyethylene (20) sorbitan monooleate], and the surfactants commercialised under the name of Span® (origin: ICI, England), such as Span® 20 (sorbitan monolaurate), Span® 40 (sorbitan monopalmitate), Span® 60 (sorbitan monostearate), and Span® 80 (sorbitan monooleate). One can further mention Cremophor® RH40 and RH60 (origin: BASF AG, Germany, which are ethoxylated hydrogenated ricin oils), Genapol® [origin: Hoechst AG, Germany, a sodium (alcohol polyglycol ether) laurylsulfate], the surfactant known under the name of Poloxamer® 407 (a diblock copolymer of ethyleneoxyde and propyleneoxyde, also commercialised under the names of Pluronic® F 127 and Pluracare® F 127, origin: BASF AG, Germany); Tetronic® (origin: BASF AG, Germany); DC 3225 C, DC 5200, DC 193 (origin: Dow Corning, USA); Abil® Em 97 (origin: Goldschmidt).

The perfuming ingredients that can be used in the present invention are all the ingredients commonly used in perfumery. These ingredients shall not be described in greater detail here, as their description cannot be exhaustive and the skilled person is able to choose them using his general knowledge and as a function of the desired olfactory effect. These perfuming ingredients belong to a variety of chemical classes, as varied as alcohols, aldehydes, ketones, esters, ethers, acetates, nitriles, terpernic hydrocarbons, heterocyclic nitrogen- or sulfur- containing compounds, as well as essential oils of natural and synthetic origin. Many of these ingredients are furthermore described in reference textbooks such as the book of S. Arctander, Perfume and Flavour Chemical, 1969, Montclair, New Jersey, USA, or its more recent versions, or other books of similar nature. The nature of the perfuming ingredients is not an essential parameter of the invention, and

the choice of the latter shall be solely dictated by hedonic requirements, i.e. as a function of the fragrance or odorant effect that it is desired.

The emulsion of the invention can be easily prepared by conventional mixing and homogenising methods, which therefore do not require a more detailed description here.

According to the present invention, emulsions are created with an average drop size above 200 nm, preferably below 1 μm .

The invention will now be illustrated by the following non-restrictive examples, in which the temperatures are indicated in degrees Celsius, the proportions of the ingredients are given in % by weight and the abbreviations have the usual meaning in the art.

Embodiments of the Invention

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Example 1

Preparation of an alcohol-free perfuming composition in the form of a water-in-oil emulsion

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A water-in-oil emulsion containing a perfuming base has been prepared with the below-specified ingredients. Incorporation of the fluorinated oil was done after emulsification of the other ingredients by a current method in the art.

| 25 | Ingredients | Parts by weight |
|----|---|-----------------|
| | Perfuming base * | 10.00 |
| | Silicon DC [®] 345 ¹⁾ | 43.00 |
| | Methoxynanofluorobutane 2) | 15.00 |
| | Water | 16.45 |
| 30 | 1,2-Butanediol | 11.55 |
| | Abil® Em 97 3) | 4.00 |
| | Total | 100.00 |

1) origin: Dow Corning

2) origin: 3M

3) origin: Goldschmidt

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* The perfuming base was obtained by mixing the following ingredients :

| | Ingredients | Parts by weight |
|----|---|-----------------|
| | Citronellyl acetate | 3 |
| 10 | Geranyl acetate | 9 |
| | Linalyl acetate | 276 |
| | 10% * Aldehyde C10 | 3 |
| | 10% * Aldehyde C12 | 12 |
| | Methyl anthranilate | 16 |
| 15 | Bergamot essential oil | 226 |
| | Cetalox ® 1) | 5 |
| | Lemon essential oil | 318 |
| | Dihydromyrcenol ²⁾ | 60 |
| | Dipropyleneglycol | 20 |
| 20 | 10% * Elemi ³⁾ | 20 |
| | Fleuria 41063 B 4) | 3 |
| | Ethyl linalol | 66 |
| | 10% * 3-(4-Methoxyphenyl)-2-methylpropanal 4) | 30 |
| | Geraniol | 6 |
| 25 | 50% * Habanolide ® 5) | 130 |
| | Hedione ^{® 6)} | 215 |
| | Hedione [®] HC ⁷⁾ | 72 |
| | 10% ** Indol | 12 |
| | Iso E Super ⁸⁾ | 85 |
| 30 | Lavandin grosso essential oil | 26 |
| | 1% * Liffarome ^{® 9)} | 20 |
| | Linalol | 40 |
| | Sfuma madarin essential oil | 5 |

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| | 10% * Crinkled mint essential oil | 30 |
|----|---------------------------------------|------|
| | Bigarade Neroli essential oil | 130 |
| | Portugal Florida orange essential oil | 80 |
| | Phenethylol | 9 |
| 5 | Petitgrain essential oil | 63 |
| | Pipol | 5 |
| | Rosemary essential oil | 16 |
| | Terpineol | 9 |
| | Violet essential oil | 50 |
| 10 | 1% * Zestover ¹⁰⁾ | _30 |
| | Total | 2100 |

- * in dipropyleneglycol (DIPG)
- ** in triethanolamine
- 15 1) 8,12-epoxy-13,14,15,16-tetranorlabdane
 - 2) origin: International Flavours and Fragrances, USA
 - 3) 3-allyl-1,2,3-trimethoxybenzene; origin: Calchauvet, Grasse, France
 - 4) origin: Firmenich SA, Geneva, Switzerland
 - 5) pentadecenolide; origin: Firmenich SA, Geneva, Switzerland
- 20 6) Methyl dihydojasonate; origin: Firmenich SA, Geneva, Switzerland
 - 7) Methyl dihydrojasmonate with high content of isomer cis; origin: Firmenich SA, Geneva, Switzerland
 - 8) 1-(octahydro-2,3,8,8-tetramethyl-2-naphtalenyl)-1-ethynone; origin: International Flavours and Fragrances, USA
- 25 9) 3-hexenyl methyl carbonate; origin: International Flavours and Fragrances, USA
 - 10) 9-decen-1-ol; origin: International Flavours and Fragrances, USA

The density of each of the two phases of the composition was measured at 30° on a density meter (DMA 4500; Anton Paar). The density of the aqueous phase was 1.0209 and that of the oily phase was 1.0249, thus forming a stable transparent emulsion.

Moreover, the respective refractive index of each phase was also measured and the difference between the two phases was of 0.0058.

Example 2

Preparation of an alcohol-free perfuming composition in the form of a water-in-oil emulsion

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A water-in-oil emulsion containing a perfuming base has been prepared with the below-specified ingredients. Incorporation of the fluorinated oil was done after emulsification of the other ingredients by a current method in the art.

| 10 | Ingredients | Parts by weight |
|----|---|-----------------|
| | Perfuming base * | 10.00 |
| | Silicon DC [®] 345 ¹⁾ | 45.00 |
| | Methoxyfluorobutane 2) | 13.00 |
| | Water | 15.15 |
| 15 | 1,2-Butanediol | 12.85 |
| | Abil [®] Em 97 ³⁾ | 4.00 |
| | Total | 100.00 |

1) origin: Dow Corning

20 2) origin: 3M

3) origin: Goldschmidt

* The perfuming base was obtained by mixing the following ingredients:

| $^{\circ}$ | 4 |
|------------|---|
| L | _ |

| | Ingredients | Parts by weight |
|----|------------------------|-----------------|
| | Benzyl acetate | 250 |
| | Pipol acetate | 70 |
| | Styrallyl acetate | 230 |
| 30 | Phenylacetic aldahyde | 10 |
| | Ambrettolide ® 1) | 10 |
| | Astrotone | 300 |
| | Bergamot essential oil | 1160 |

| | β-Ionone | 550 |
|----|---|------|
| | Cassis essential oil | 150 |
| | 50% * Cetalox ^{® 2)} | 60 |
| | Lemon essential oil | 850 |
| 5 | Citronellol | 210 |
| | Damascenone | 20 |
| | 4-Decanolide | 20 |
| | Dihydromyrcenol ³⁾ | 440 |
| | Dipropyleneglycol | 20 |
| 10 | Ethyl linalol | 720 |
| | 7-Methyl-2H,4H-1,5-benzodioxepin-3-one 4) | 100 |
| | Floralozone ^{® 5)} | 50 |
| | 3-(4-Methoxyphenyl)-2-methylpropanal 4) | 170 |
| | Fructone ^{® 6)} | 100 |
| 15 | Galbex ^{® 4)} | 50 |
| | γ-Damascone | 5 |
| | Geranium essential oil | 30 |
| • | Grapefruit essential oil | 100 |
| | Habanolide ^{® 7)} | 1120 |
| 20 | Hedione ® 8) | 2890 |
| | Hedione ® HC 9) | 950 |
| | Heliopropanal 10) | 400 |
| | Indol | 35 |
| | Iso E Super 11) | 380 |
| 25 | Lavender grosso essential oil | 40 |
| | Liffarome ® 12) | 1 |
| | Lilial ^{® 13)} | 1050 |
| | Lyral ^{® 14)} | 430 |
| | Sfuma mandarin essential oil | 270 |
| 30 | Melonal 15) | 3 |
| | Crinkled mint essential oil | 20 |
| | Peony 434017 4) | 60 |
| | Peony white HS 100001 4) | 200 |
| | Phenethylol | 80 |

| | Phenylhexanol | 50 |
|---|---------------------------|-------|
| | Pipol | 20 |
| | Orange essential oil | 500 |
| | Rosalva ¹⁶⁾ | 4 |
| 5 | Benzyl salicylate | 400 |
| | Pipol salicylate | 400 |
| | 10% ** BHT ¹⁷⁾ | 200 |
| | Zestover 18) | 22 |
| | Total | 15200 |

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- * in 2-(2-ethoxyethoxy)-1-ethanol
- ** in propyleneglycol
- 1) origin: Givaudan-Roure SA, Vernier, Switzerland
- 2) 8,12-epoxy-13,14,15,16-tetranorlabdane; origin: Firmenich SA, Geneva,
- 15 Switzerland
 - 3) origin: International Flavours and Fragrances, USA
 - 4) origin: Firmenich SA, Geneva, Switzerland
 - 5) 3-(4-ethylphenyl)-2,2-dimethylpropanal + 3-(2-ethylphenyl)2,2-dimethylpropanal; origin: International Flavours and Fragrances, Switzerland
- 20 6) 2-methyl-1,3-dioxalane-2-ethylacetate; origin: International Flavours and Fragrances, USA
 - 7) pentadecenolide; origin: Firmenich SA, Geneva, Switzerland
 - 8) methyl dihydrojasmonate; origin: Firmenich SA, Geneva, Switzerland
 - 9) methyl dihydrojasmonate with high content of isomer *cis*; origin: Firmenich SA, Geneva, Switzerland
 - 10) 3-(1,3-benzodioxol-5-yl)-2-methylpropanal; origin: Firmenich SA, Geneva, Switzerland
 - 11) 1-(octahydro-2,3,8,8-tetramethyl-2-naphtalenyl)-1-ethanone; origin: International Flavours and Fragrances, USA
- 30 12) 3-hexenyl-methyl carbonate; origin: International Flavours and Fragrances, USA
 - 13) 3-(4-tert-butylphenyl)-2-methylpropanal; origin: Givaudan-Roure SA, Vernier, Switzerland

- 14) 4-(4-hydroxy-4-methylpentyl)-3-cyclohexen-1-carbaldehyde + 3-(4-hydroxy-4-methylpentyl)-3-cyclohexene-1-carbaldehyde; origin: International Flavours and Fragrances, USA
- 15) 2,6-dimethyl-5-heptanal; origin: Givaudan-Roure SA, Vernier, Switzerland
- 5 16) 9-decen-1-ol; origin: International Flavours and Fragrances, USA
 - 17) 2,6-di-tert-butyl-4-hydroxytoluene
 - 18) 2,4-dimethyl-3-cyclohexene-1-carbaldehyde; origin: Firmenich SA, Geneva, Switzerland
- The density of each of the two phases of the composition was measured at 30° on a density meter (DMA 4500; Anton Paar). The density of the aqueous phase was 1.0193 and that of the oily phase was 1.0218, thus forming a stable transparent emulsion.
- Moreover, the respective refractive index of each phase was also measured and the difference between the two phases was of 0.0086.

Claims

- 1. Alcohol free perfuming composition in the form of a transparent water-inoil or oil-in-water emulsion susceptible of being sprayed, comprising an aqueous phase and an oily phase containing at least 3% by weight of perfuming ingredients relative to the total weight of the composition, wherein the difference between the density of the aqueous phase and that of the oily phase is less than or equal to 0.005.
- 2. Perfuming composition according to claim 1, wherein the difference between the density of the oily phase and that of the aqueous phase in less than or equal to 0.003.
- 3. Perfuming composition according to claim 1, characterised in that the viscosity of the emulsion is below 10 Pa.s.
 - 4. Perfuming composition according to claim 1, in the form of a water-in-oil emulsion.
- 5. Perfuming composition according to claim 4, comprising from 50 to 95% by weight of oily phase and from 5 to 50% by weight of aqueous phase.
- 6. Perfuming composition according to claim 1, characterised in that the oily phase of the emulsion comprises from 5 to 50% by weight of a volatile fluorinated oil having a density higher than 1.
 - 7. Perfuming composition according to claim 6, characterised in that the volatile fluorinated oil is selected from the group consisting of hydrofluoroethers.
- 30 8. Perfuming composition according to claim 7, characterised in that the volatile fluorinated oil is methoxynanofluorobutane.
 - 9. Process for the preparation of a perfuming composition according to any one of claims 1 to 8, characterised in that a volatile fluorinated oil having a density higher

than 1 is added to the oily phase of the emulsion after an emulsification with the aqueous phase of the emulsion.

10. Use of a volatile fluorinated oil having a density higher than 1 as a stabilisation agent of a water-in-oil or oil-in-water emulsion.

Abstract

A particularly stable perfuming composition in the form of a transparent fluid water-in-oil or oil-in-water emulsion is obtained by addition of a substance to the oily phase, capable of bringing closer the densities of the respective dispersed and continuous phases.